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COMPLETE SPECIFICATION

Ionic Bright-Drying Polish Emulsions and process for their Manufacture

We, FARBWERKE HOECHST AKTIENGESELLSCHAFT, vormals Meister Lucius & Brüning, a Body Corporate recognised under German Law, of 6230 Frankfurt (M)-Hoechst, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to ionic bright-drying polish emulsions which are preferably free from olein and to a process for their manufacture.

It has been proposed to use ionic bright-drying polish emulsions based on natural or synthetic ester waxes for treating floors. As emulsifiers there have been proposed the soaps of olein in admixture with volatile amines.

It has also been proposed to replace the ester waxes in bright-drying polish emulsions wholly or partially by polyethylene waxes. However, polyethylene waxes are hydrocarbon waxes that do not contain functional groups and so cannot be emulsified in water. Emulsifiable modified polyethylene waxes are obtained either by the oxidation of the non-emulsifiable types, or by the copolymerization of ethylene with one or more partially or totally neutralized, unsaturated organic acids as described, for example, in British Patent Specification 929,643. Wax films of these copolymers have the advantage that they do not yellow on being exposed to the air as do films of oxidized polyethylene waxes.

The non-emulsifiable polyethylene waxes, their emulsifiable oxidates and the aforesaid emulsifiable ethylene copolymers are all softer than either natural or synthetic ester waxes. It has hitherto only been possible to emulsify the latter two products by the addition of olein, and since a content of olein reduces the hardness of wax films, bright-drying polish films obtained from polyethylene waxes generally have a low resistance to absorbing dirt and sustaining damages.

Polyethylene waxes are, therefore, generally not worked up as such but in mixture with ester waxes to give emulsions. Although there is a great demand for bright-drying polish emulsions on the basis of polyethylene waxes which are free from olein and yield hard films, such emulsions have not been described hitherto.

The present invention provides a process for the manufacture of ionic bright-drying polish emulsions wherein a copolymer, obtained by the polymerization of from 99 to 50 per cent by weight of ethylene and from 1 to 50 per cent by weight of crotonic acid at a temperature within the range of from 50°C to 250°C and a pressure within the range of from 200 to 950 atmospheres gauge in the presence of a free radical-forming compound and in an organic solvent, is emulsified in water in the presence of a base.

These copolymers that can be obtained according to British Patent No. 1,098,740 (Application No. 57647/66) possess free carboxyl groups which aid emulsification of the copolymer or which may be used for further chemical reactions. There are preferably applied polymers prepared as above which have a content of crotonic acid of from 3 to 30 per cent by weight, calculated on the copolymer, whose melting points are within the range of from 60°C to 115°C and which have melt viscosities (measured at 120°C in a capillary viscosimeter according to Ubbelohde) within the range of from 500 to 10,000 Centistokes. These polymers are colourless, odourless, wax-like substances which are preferably incorporated into the bright-drying polish emulsions of

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the invention in an amount within the range of from 10 to 15 per cent by weight, calculated on the total amount of the emulsion. The copolymers are incorporated into the dispersion in known manner by a water-in-wax or a wax-in-water process.

For the preparation of the emulsions in accordance with the present invention, there is preferably used as base an amine or an amino alcohol. However, there may occasionally be used a concentrated aqueous solution of 40 to 50 per cent strength of an alkali, for example, a solution of potassium hydroxide. As amine or amino alcohol there may be used, for example, an aliphatic or cycloaliphatic primary, secondary or tertiary amine or amino alcohol that contains from 2 to 6 carbon atoms per aliphatic or cycloaliphatic group, for example, diethyl amine, morpholine, 3-methoxypropylamine, diethylamino-ethanol, 2-amino-2-methylpropanol or triethanolamine. The base is preferably present in an amount of from 2 to 5 per cent by weight calculated on the total amount of the polyethylene wax emulsion. If the emulsification is carried out using an amine as base, a bright-drying film that has a special resistance to water is obtained after some time, as a result of the volatility of the amine. If an inorganic base is used, the alkali remains in the film and the resistance to water is reduced. In some cases it is recommended, however, to add slight amounts of an alkali in addition to amines to obtain especially finely dispersed emulsions. Additions of up to 0.5 per cent by weight of alkali, calculated on the total amount of the emulsion, do not impair the resistance of the polish to water.

The bright-drying polish emulsions according to the present invention, prepared in the usual manner by a wax-in-water or water-in-wax process, can be mixed with conventional copolymer dispersions, for example, styrene/acrylate copolymers, and other known additives, for example, resins soluble in alkalies, or wetting or levelling agents.

The polish emulsions according to the present invention are preferably obtained completely or substantially free from olein.

The advantages of using bright-drying polish emulsions that are free from olein rather than olein-containing bright-drying polish emulsions on the basis of polyethylene waxes are shown most clearly by testing the bright-drying polish films as to their absorption of dirt when being walked on. The following Table shows the results of such a two-week test on light-coloured PVC plates that have been treated with conventional bright-drying polish emulsions containing olein as well as bright-drying polish emulsions according to the present invention that are free from olein, and it can be seen that the penetration number (PN) in the case of the olein-containing emulsions is noticeably higher than in the case of the emulsions according to the present invention and that, consequently, the absorption of dirt is correspondingly higher.

Tests 1 and 2 in the following Table were for comparison purposes and were conducted using polyethylene wax emulsions that contained olein. These emulsions were prepared as follows:

Emulsion 1 used in Test 1: 2.0 Parts by weight of morpholine and 2.0 parts by weight of olein were stirred at 120°C into 12.0 parts by weight of a molten polyethylene wax prepared according to Example 5 of British Patent Specification 929,643. The melt was introduced into 84.0 parts by weight of boiling water, while stirring vigorously, the mixture was cooled rapidly and filtered.

The basic emulsion was further mixed, as described in Example 1 hereinafter, in order to acquire the optimum wetting and levelling properties required for practical application.

Emulsion 2 used in Test 2: The emulsion was prepared from 12.0 parts by weight of polyethylene wax (Hoechst Wachs PAD 521), 1.0 part by weight of olein, 1.3 parts by weight of amino methyl propanol and 85.7 parts by weight of water in a manner analogous to that described above.

The basic emulsion was further mixed as described in Example 2 hereinafter.

TABLE

Test No.	Composition of Polish Emulsion	Gloss on drying of the film prior to being tested by being walked on (1)	Absorption of dirt (2)	Penetration Number (PN) of the emulsion body (3)
1 (for comparison)	Basic emulsion 1 mixed according to Example 1	57	6	18.5
2 (for comparison)	Basic emulsion 2 mixed according to Example 2	55	4 — 5	7.5
3	Basic emulsion according to Example 1, mixed according to Example 1	63	3	3
4	Basic emulsion according to Example 1, mixed according to Example 2	50	3	3

1) The gloss values were photoelectrically measured by means of the gloss-meter according to Dr. B. Lange, Berlin. The values are percentage figures referring to a certain gloss standard (100%).

2) The absorption of dirt was determined by subjective judgement according to a scale ranging from 1 to 6, 1 meaning a low and 6 a high absorption of dirt.

3) The penetration number (PN) was determined according to Richardson ASTM D 1321—57/DIN (German Industrial Standards) 1995 and designates the depth of penetration of a standardized needle into the wax.

The following Examples illustrate the invention, the parts and percentages being by weight:

EXAMPLE 1

At 120°C, 3.0 parts of diethyl aminoethanol were slowly stirred into 12.0 parts of a molten copolymer of ethylene and crotonic acid (proportion of crotonic acid 6.3%, crystallization point 99.5°C) obtained according to the method described in Example 2 of British Patent Application No. 57647/66 (serial No. 1,098,740) by the copolymerization of crotonic acid and ethylene at a temperature of 140°C and a pressure of 500 atmospheres gauge in the presence of azobisisobutyronitrile. As soon as the amine was incorporated, the mixture was introduced into 85.0 parts of boiling water while stirring vigorously, and the mixture was rapidly cooled to room temperature and filtered. The basic emulsion obtained which, fundamentally, could already be used as a bright-drying polish emulsion, was further mixed as indicated hereunder in order to acquire the optimum wetting and levelling properties required for practical application: 28.5 parts of the aforesaid emulsion were mixed with 57 parts of a conventional copolymer dispersion of styrene/acrylate (VPW 5403 D/1/Farbwerke Hoechst), 14.5 parts of an ammoniacal solution (of 15% strength) of a colophonium/maleate resin (Shanco 334 of Shanco Plastics and Chemicals Inc.), 1.5 parts of ethyl diglycol, 0.4 part of tributoxy ethyl phosphate (KP 140 of Food Machinery and Chemical Corp., New York) and 0.4 part of a wetting agent on the basis of perfluorinated carboxylic acids ("FC 128" of 3-M Company). The properties of a bright-drying polish film produced with this emulsion are shown in Test 3 of the above Table.

EXAMPLE 2

70 Parts of the basic emulsion prepared according to Example 1 were mixed with 30 parts of a styrene/acrylate copolymer dispersion, 10 parts of an ammoniacal solution (of 15% strength) of Shanco resin, 0.5 part of

tributoxy ethyl phosphate and 1.0 part of the wetting agent "FC 128". The properties of the bright-drying polish film prepared from this mixture were found to be as shown in Test 4 of the above Table.

WHAT WE CLAIM IS:—

1. A process for the manufacture of an aqueous ionic bright-drying polish emulsion, wherein a copolymer, obtained by the copolymerization of from 99 to 50 per cent by weight of ethylene and from 1 to 50 per cent by weight of crotonic acid at a temperature within the range of from 50°C to 250°C and a pressure within the range of from 200 to 950 atmospheres gauge in the presence of a free-radical polymerization initiator and in an organic solvent, is emulsified with water in the presence of a base.

2. A process as claimed in claim 1, wherein the copolymer contains from 3 to 30 per cent by weight of crotonic acid, calculated on the weight of the copolymer, and has a melting point within the range of from 60°C to 115°C and a melt viscosity (measured at 120°C in a capillary viscosimeter according to Ubbelohde) within the range of from 500 to 10,000 Centistokes.

3. A process as claimed in claim 1 or claim 2, wherein the emulsion contains from 10 to 15 per cent by weight, calculated on the total amount of the emulsion, of the copolymer.

4. A process as claimed in any one of claims 1 to 3, wherein the base is present in an amount of from 2 to 5 per cent by weight, calculated on the total amount of the emulsion.

5. A process as claimed in any one of claims 1 to 4, wherein the base is an amine or an amino alcohol.

6. A process as claimed in claim 5, wherein the base is an aliphatic or cycloaliphatic primary, secondary or tertiary amine or amino alcohol that contains from 2 to 6 carbon atoms per aliphatic or cycloaliphatic group.

7. A process as claimed in claim 6, wherein the base is diethyl amine, morpholine, 3-methoxy-propylamine, diethylamino ethanol, 2-amino-2-methylpropanol or triethanolamine.

8. A process as claimed in any one of claims 1 to 7, wherein as base there is used an amine or an amino alcohol and up to 0.5 per cent by weight, calculated on the total amount of the emulsion, of an alkali.

9. A process as claimed in any one of claims 1 to 4, wherein the base is an aqueous solution of an alkali of from 40 to 50 per cent strength.

10. A process as claimed in claim 9, wherein the alkali is potassium hydroxide.

11. A process as claimed in any one of claims 1 to 10, wherein the resulting polish emulsion is completely or substantially free from olefin.

12. A process as claimed in any one of claims 1 to 11, wherein the emulsion is mixed with a copolymer dispersion or a resin that is soluble in alkaline solutions.

13. A process as claimed in any one of claims 1 to 12, wherein the emulsion is mixed with a wetting or levelling agent.

14. A process as claimed in claim 1, substantially as described in Example 1 or Example 2 herein.

15. An aqueous ionic bright-drying polish emulsion comprising from 10 to 15 per cent by weight, calculated on the total amount of the emulsion, of a copolymer obtained by the copolymerization of from 99 to 50 per cent by weight of ethylene and from 1 to 50 per cent by weight of crotonic acid at a temperature within the range of from 50°C to 250°C and a pressure within the range of from 200 to 950 atmospheres gauge in the presence of a free-radical polymerization initiator and in an organic solvent, and from 2 to 5 per cent by weight, calculated on the total amount of the emulsion, of a base.

16. An aqueous ionic bright-drying polish emulsion whenever obtained by a process as claimed in any one of claims 1 to 14 herein.

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